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## PATENT SPECIFICATION



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### PROVISIONAL SPECIFICATION

#### A Process for Producing Surface Tension-reducing Substances in a Solid Form

I, JAMES GEORGE FIFE, M.Sc. (Lond.), F.I.C., Consulting Chemist & Chartered Patent Agent, of 20 to 23, Holborn, London, E.C.1, a British subject, do hereby declare the nature of this invention (as communicated to me by N. V. De Bataafsche Petroleum Maatschappij of 30 Carel van Bylandtlaan, The Hague, Holland, a Body Corporate organised under the Laws of Holland) to be as follows:—

This invention relates to a process for producing in a solid form substances which reduce the surface tension of water and aqueous solutions hereinafter referred to as "surface tension-reducing substances", such as washing and wetting agents and the like.

It is particularly applicable to such surface tension-reducing substances as cannot be obtained or are difficult to obtain in solid form by evaporation in the normal way on account of their low decomposition temperature.

The invention will be further explained below with reference to the production, in a solid form, from solutions of salts, such as the alkaline salts, of acid sulphuric acid alkyl esters obtained by reaction between liquid alkenes or alkene-containing mixtures, preferably such as have about 10 to 18 carbon atoms in the molecule, and sulphuric acid.

According to the process of the present invention the substances referred to are obtained in the desired form by atomising their solutions into hot gases, which is known per se, and subsequently compressing the dry powders thus obtained, whereby these powders are moulded into dense aggregates. If so desired, the powders may be mixed with other substances, e.g. with an inert filler, before compression.

The powders obtained by the atomisation, which also contain the inorganic salts present in the solutions as well as any other components and which further as a rule contain less than about 0.5% by weight of water, are found on microscopic examination to consist of hollow spherical bodies. Consequently they

have a very low apparent specific gravity, viz. about 0.1 or lower.

If these and similar light powders of surface tension-reducing substances or light powders containing such substances are used for washing, wetting and similar purposes and are with this object dissolved in water, difficulties arise, because the powders float on the surface of the water. This is also fostered by the foam formed, if it is desired to promote mixing by stirring, the powder then being pressed upwards by the foam. Additional drawbacks of these very light powders are that they readily scatter in the air and involve high packing and transport costs.

Now, according to the present invention the powders are compressed under high pressure and subsequently reground, powders with an appreciably higher apparent specific gravity e.g. an apparent specific gravity of about 0.8, being thus obtained. These powders are much easier to dissolve and scatter less readily in the air and involve lower packing and transport costs.

If it is desired to obtain particularly heavy powders, it is advisable to repeat the compression and the grinding one or more times.

The process according to the invention, however, is not restricted to the production of heavier from lighter powders via compression and grinding, but is concerned generally with the compression to dense aggregates of the light powders obtained in the atomisation. These aggregates may as such have attained a final form, e.g. the form of grains or smaller blocks, or may be converted into another form, e.g. by compressing grains to blocks or by reducing larger aggregates to flakes. It is not advisable to compress the light powder obtained in the atomisation directly into large compact blocks, as this will give rise to difficulties in discharging on account of the material adhering to the die and the bottom of the press. This difficulty may be obviated by first compressing the light powder in, for example, continuous presses to grains or

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strings and subsequently moulding this material into blocks in die presses.

The compression of the light powder may be effected on rollers, in eccentric presses, hydraulic presses, continuous screw presses, or the like. It is advisable in the compression of the light powder to strings or grains to use a short compression chamber with a short worm, rendering it possible for the enclosed air to escape as soon as possible, so that an increase of the temperature to the decomposition temperature as a result of the compression of the air is avoided in a simple manner. The rise in temperature may also be prevented by artificial cooling. It is, of course, also possible to

apply simultaneously combinations of such measures. The maximum admissible compression pressure is that above which the products begin to flow, which with ester salts is the case at pressures of about 400 atms. As a rule, however, the pressures will be kept considerably lower, with a view to preventing the light powders, which are usually white, becoming brown.

Dated this 8th day of August, 1939.  
ELKINGTON & FIFE,  
Consulting Chemists and  
Chartered Patent Agents,  
20 to 23, Holborn, London, E.C.1,  
Agents for the Applicant.

## COMPLETE SPECIFICATION

### A Process for Producing Surface Tension-reducing Substances in a Solid Form

I, JAMES GEORGE FIFE, M.Sc. (Lond.), F.I.C., Consulting Chemist & Chartered Patent Agent, of 20 to 23, Holborn, London, E.C.1, a British subject, do hereby declare the nature of this invention and in what manner the same is to be performed (as communicated to me by N. V. De Bataafsche Petroleum Maatschappij, of Willemstad, Curacao, Netherlands West Indies, formerly of 30, Carel van Bylandtlaan, The Hague, Holland, a Body Corporate, organised under the Laws of the Netherlands), to be particularly described and ascertained in and by the following statement:—

This invention relates to a process for producing in a solid form organic substances of high molecular weight which reduce the surface tension of water and aqueous solutions, hereinafter referred to as "surface tension-reducing substances" such as washing and wetting agents and the like used for example in the textile industry.

It is particularly applicable to such surface tension-reducing substances as cannot be obtained or are difficult to obtain in solid form by evaporation in the normal way on account of their low decomposition temperature.

The invention will be further explained below with reference to the production, in a solid form, from solutions of salts, such as the alkaline salts, of acid sulphuric acid alkyl esters obtained by reaction between liquid alkenes or alkene-containing mixtures, preferably those having 8 to 20 and more preferably 10 to 18 carbon atoms in the molecule, and sulphuric acid.

It will be understood, however, that

the invention is not limited to such compounds but may be advantageously applied to the corresponding alkaline earth, heavy metal, ammonium, aluminium and other inorganic salts as well as organic salts such, for example, as amine salts, sulphonium salts, ammonium and phosphonium and like salts of alkyl acid sulphates. Instead of secondary alkyl sulphates primary or tertiary alkyl sulphates may be treated. The acid sulphate esters involved may have cyclic groups which may be either aromatic or alicyclic or both types of groups may be present in the molecule which may or may not also contain unsaturated linkages and/or substituent groups or atoms such, for example, as hydroxy or nitro or amine or ether or ester or like groups or halogen atoms. The process may be used with the corresponding phosphate ester salts or salts of sulphonic acids or the like instead of with the foregoing sulphate ester compounds.

According to the process of the present invention the substances referred to are obtained in the desired form by atomising their solutions into hot gases, which is known per se, and subsequently compressing the dry powders thus obtained, whereby these powders are moulded into dense aggregates. If so desired, the powders may be mixed with other substances, e.g. with an inert filler, or with a modifying agent such as a higher alcohol, before atomisation or more preferably after atomisation but before compression.

The powders obtained by the atomisation, which also contain the inorganic

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- salts present in the solutions as well as any other components and which further as a rule contain less than about 0.5% by weight of water, are found on microscopic examination to consist of hollow spherical bodies. Consequently they have a very low apparent specific gravity, usually less than about 0.2 and generally about 0.1 or lower.
- 10 If these and similar light powders of surface tension-reducing substances or light powders containing such substances are used for washing, wetting and similar purposes and are with this object dissolved in water, difficulties arise, because the powders float on the surface of the water. This is also fostered by the foam formed, if it is desired to promote mixing by stirring, the powder then being pressed upwards by the foam.
- 15 Additional drawbacks of these very light powders are that they readily scatter in the air and involve high packing and transport costs.
- 25 Now, according to the present invention the powders are compressed under high pressure and if desired subsequently reground, powders with an appreciably higher apparent specific gravity, e.g. an apparent specific gravity of about 0.8, being thus obtained. These powders are much easier to dissolve and scatter less readily in the air and involve lower packing and transport costs.
- 30 If it is desired to obtain particularly heavy powders, it is advisable to repeat the compression and the grinding one or more times.
- The process according to the invention, however, is not restricted to the production of heavier from lighter powders via compression and grinding, but is concerned generally with the compression to dense aggregates of the light powders obtained in the atomisation.
- 40 These aggregates may as such have attained a final form, e.g. the form of grains or smaller blocks, or may be converted into another form, e.g. by compressing grains to blocks or by reducing larger aggregates to flakes. It is not advisable to compress the light powder obtained in the atomisation directly into large compact blocks, as this will give rise to difficulties in discharging on account of the material adhering to the die and the bottom of the press. This difficulty may be obviated by first compressing the light powder in, for example, continuous presses to grains or strings and subsequently moulding this material into blocks in die presses.
- 50 The compression of the light powder may be effected on rollers, in eccentric presses hydraulic presses, continuous screw presses, or the like. It is advisable in the compression of the light powder to strings or grains to use a short compression chamber with a short worm, rendering it possible for the enclosed air to escape as soon as possible, so that an increase of temperature to the decomposition temperature as a result of the compression of the air is avoided in a simple manner. The rise in temperature may also be prevented by artificial cooling. It is, of course, also possible to apply simultaneously combinations of such measures. The maximum admissible compression pressure is that above which the products begin to flow, which with ester salts is the case at pressures of about 400 atms. As a rule, however, the pressures will be kept considerably lower, with a view to preventing the light powders, which are usually white, becoming brown.
- 70 The pressure should be sufficient to substantially increase the apparent specific gravity of the product, most preferably sufficient to increase the apparent specific gravity to at least about 0.5 and more preferably to 0.6 to about 0.8 or more. An indicated above it is not necessary, however, that such increase be effected in a single operation and in fact it is advantageous, particularly where products of especially high apparent specific gravity are desired, to submit the light powder obtained by atomisation, to repeated compression. Thus to produce heavy powders the light spray dried products may be compressed and reground more than once.
- 80 Having now particularly described and ascertained the nature of my said invention, and in what manner the same is to be performed (as communicated to me from abroad), I declare that what I claim is:—
1. A process for producing surface tension-reducing substances in a solid form wherein solutions of such substances are atomised into hot gases and the dry powders thus obtained are compressed and moulded into dense aggregate.
  2. A process as claimed in claim 1 wherein the dense aggregates obtained are reground to produce powders.
  3. A process as claimed in claim 1 or 2 wherein the surface tension-reducing substances are acid sulphuric acid alkyl esters having from 8 to 20 carbon atoms in the molecule.
  4. A process as claimed in any one of the preceding claims wherein the compression is repeated one or more times.
  5. Aggregates of surface tension-reducing substances when produced by

the process claimed in any one of the preceding claims 1, 3 and 4.

6. Powdered surface tension-reducing substances when produced by the process claimed in any one of the preceding claims 2, 3 and 4.

Dated this 8th day of August, 1940.  
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Agents for the Applicant.

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